JOURNAL

OF THE

AMERICAN CHEMICAL SOCIETY.

VOL. VII.—NO. 1.

JANUARY.

1885.

COMMITTEE ON PAPERS AND PUBLICATIONS:

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W. RUPP, F. P. VENABLE.

CONTENTS:

	Page.
Proceedings. January meeting	1
Treasurer's Report, 1884	2
I. The Manufacture of Graphite Crucibles (second paper); by J. C. Booth, Ph. D.	4
II. Note on Camphor Motions; by P. Casamajor	13
III. On Methods of Indigo Testing; by H. M. Rau, Ph. D.	16
IV. A new form of Dropping Flask; by F. V. Pool	20
V. Note on Oil of Malt; by Narcisse Pigeon	22
Abstracts from Chemical Journals	
General and Inorganic Chemistry	23
Organic Chemistry	29
Analytical "	32
Abstracts of American Patents relating to Chemistry	34

PUBLISHED MONTHLY.

NEW YORK:

JOHN POLHEMUS,

102 Nassau Street.

1885.

The **Journal of the American Chemical Society** appears monthly, except during July and August. It will contain all papers read before the Society, as well as abstracts from American and Foreign Journals, and a list of American Patents relating to Chemical Industries.

The Price of Subscription is $5.00 per year, in advance.

The Meeting Room and Library of the Society are at Room 1, University Building, Washington Square, New York.

The Regular Monthly Meeting is held on the first Friday of every month, at 8 P. M.

Corresponding Secretary:

P. CASAMAJOR, Care of Havemeyer & Elder, Brooklyn, E. D.

Treasurer:

T. O'CONNOR SLOANE, 119 Pearl St., New York.

The Library will be open to Members regularly on Fridays, from 6 to 10 P. M., and at any other time on presenting to the Janitor of the University Building a ticket, which can be obtained of the Librarian.

Back numbers of the Journal are sold to Members at $3.00, to others at $5.00, per volume. Apply to the Librarian

W. RUPP, 117 Pearl St., New York.

The Committee on Papers and Publications respectfully call attention to the resolutions of the Society, requiring that papers be submitted to the Committee before being read.

Members who cannot be present at the meetings, are requested to send their papers in care of the Committee, who can guarantee prompt publication of all articles accepted.

Authors will be furnished, gratis, with 20 reprints of each article published in the Journal, if application for the same be made when the article is sent.

Members are requested to notify the Committee of any change of address, or failure in the delivery of the Journal. All communications relating to the Journal should be addressed in care of the Editor

A. A. BRENEMAN, 97 Water St., New York.

PROCEEDINGS OF THE AMERICAN CHEMICAL SOCIETY.

REGULAR MEETING, held January 2d, 1885.

Meeting called to order at 8.30 P.M.; Dr. A. R. Leeds in the Chair.

The minutes of the meeting of December 5th, 1884, were read and approved.

REGULAR MEMBERS ELECTED.

William Elliott, 197 Pearl St., New York.

Thomas M. Morgan, Crompton, Rhode Island.

Dr. G. W. Lehmann, 57 S. Gay St., Baltimore, Md.

W. French Smith, 235 Washington St., Boston, Mass.

Walter H. Kent, Ph.D., Jamestown, New York.

Elias H. Bartley, M.D., 401 Pacific St., Brooklyn, N. Y.

A. Howard Van Sinderen, Ph.B., 45 William St., New York.

Milton R. Wood, care of Church & Co., Station G., Brooklyn, N. Y.

John T. Williams, East River Chemical Works, Cotton Exchange, Hanover Square, New York.

NOMINATIONS FOR REGULAR MEMBERSHIP.

Dr. A. Miller Jacobs, 92 New Church St., New York City.

Joseph Little, Passaic, N. J.

Edward K. Dunham, Ph.B., 93 Boylston St., Boston, Mass.

A paper by Dr. J. C. Booth, on Graphite or Black Lead Crucibles (2d paper), was read by title.

Dr. H. M. Rau read a paper on methods of Indigo Analysis.

Mr. P. Casamajor read some further notes on the Motions of Camphor on Water; being a continuation of a paper on the same subject read before the Society October 4th, 1877.

Mr. F. V. Pool described a new form of Dropping Flask.

Mr. Casamajor presented a note on Oil of Malt, by Narcisse, Pigeon.

Dr. Waller drew attention to the notice in the *Chemical News* of Dec. 5, relating to the family of Henry Watts.

Mr. Casamajor suggested that the article be reprinted and distributed with the next issue of the JOURNAL.

Dr. Waller was requested by the Society to take charge of subscriptions which might be received in response to this notice.

The Meeting was adjourned at 10.30 P. M.

C. E. MUNSELL, Recording Secretary.

TREASURER'S REPORT.

JANUARY 8th, 1885.

DR.			CR.		
1884.			1884.		
Jan.	1 To cash on hand	$198 91	Jan.	31. By Bills Paid	$199 75
Jan.	31. To collections	110 00	Feb.	29. " "	5 00
Feb.	29. "	89 50	March	31. " "	235 21
March	31. "	264 50	April	30. " "	137 32
April	30. "	191 00	May	31. " "	209 06
May	31. "	44 97	June	30. " "	101 34
June	30. "	180 50	July	31. " "	193 79
July	31. "	176 00	Aug.	31. " "	111 75
Aug.	31. "	48 00	Sept.	30. " "	3 42
Sept.	30. "	10 00	Oct.	31. " "	87 50
Oct.	31. "	75 00	Nov.	30. " "	118 73
Nov.	30. "	192 50	Dec.	31. " "	152 68
Dec.	31. "	113 00			
		$1,698 88			
				Balance	$138 33
1885.					
Jan.	1. To Cash on hand	$138 33			$1,693 88

DETAILED STATEMENT OF EXPENDITURES.

Treasurer's expenses	$30 10
Recording Secretary's expenses	54 00
Corresponding Secretary's expenses	20 50
Librarian's expenses	21 05
Rent, Janitor, and Coal	450 75
Journal	964 17
Insurance	6 00
Gas	8 98
	$1,555 55

DETAILED STATEMENT OF RECEIPTS.

Dues collected from members	$1,146 11
Advertisements	153 00
Subscriptions to Journal	44 36
Extra numbers of Journal	31 50
Donations—William Weightman	50 00
Jas. C. Booth	50 00
Albert R. Leeds	20 00
	$1,404 97

COMPARISON BETWEEN YEARS 1883 AND 1884.

	1883.	1884.
Receipts	$898 20	$1,494 97
Expenditures	832 21	1,555 55
Excess of receipts over expenditures	66 81	
Deficiency		61 58

Respectfully submitted,

T. O'CONOR SLOANE,

Treasurer.

THE MANUFACTURE OF BLACK LEAD CRUCIBLES.

By James C. Booth, Ph. D.

[Concluded from Page 283, Volume VI.]

Pulverizing the Graphite.—The manner of grinding graphite being of considerable influence on the quality of the crucible, some details upon the subject will be proper and useful. The larger masses of graphite being roughly broken by hammer into smaller pieces, the whole mixture of pieces and fine stuff is fed into an ordinary bark mill, consisting of two cast iron inverted cones, the smaller with iron teeth on its outer surface, revolving inside the larger one, similarly armed on its inner surface. The lumps are caught and crushed between these cones, and the resulting coarse and fine powder, being immediately passed between ordinary mill stones, is ground to a more uniform and finer powder, which is then assorted by sieves into finer and coarser portions, the latter being again ground and sifted until the requisite fineness is attained.

I have nothing to suggest to improve the machinery or mode of grinding, but as I differ from crucible makers in regard to the degree of fineness, I here present my views and the reasons for them. To economize time the ground graphite, passing through the sieves and consisting of fine flour on one extreme and on the other of flakes that can be split by a knife or ground to thinner scales, is usually mixed directly with the wet clay.

I regard the coarser flakes of insufficiently ground graphite as positively objectionable, because they lessen the strength of the crucible, which is the supreme consideration. I have often observed the surfaces of spalls split off from a crucible while in use, and noticed on them graphite scales of $\frac{1}{8}$ inch or more in diameter, with a part of the same scales remaining on the crucible and in the same spot. The knife easily separated more scales from the corresponding locality on spall and crucible. The facility of splitting offered by these graphite scales increases the facility with which pieces of the crucible itself split off through the feebly adhering laminations of the graphite. I have often seen a half dozen such scaling plates of graphite on a spalled surface of 2 to 4 square inches, and instantly read the cause of spalling and of serious injury to the crucible. If those coarser and thicker plates of graphite had been

ground, spalling would not have occurred. Graphite is a very feeble substance in the plane of its lamination but strongly resists a force at right angles to this plane, and, what is more extraordinary, it will do so at a white heat.

Hence black lead should be ground exceedingly fine, not leaving a plate of it more than $\frac{1}{20}$ to $\frac{1}{50}$ inch in diameter. I have tried crucibles made of black lead powder that would not quite pass through a 126 wire sieve, and found them excellent. The dust that passed through such a sieve consisted of about 75 pr. ct. of good and tough flakes, and 25 pr. ct. of a fine black powder, without apparent structure, consisting of siliceous sand and iron pyrites. "If fine grinding were adopted," the economic manufacturer asks, "What should be done with the fine powder passing the 125 wire sieve?" About $\frac{1}{8}$ of it, being siliceous, is not hurtful, but the $\frac{1}{8}$ iron pyrites is injurious, and although it is so exceeding small in quantity, yet it would be preferable to utilize it in one of three ways: 1. To separate it by a still finer sieve into good flaky graphite, to be used with the rest, and dirt, to be thrown away; or, 2. To use it in mixture with good material for covers, etc.; or 3. To sell the whole mass passing the 125 sieve, with other inferior residues, for stove blacking, &c. In such case it may sell for half the value of good black lead.

Clay.—Admirable as are the qualities of black lead to resist the action of a white heat, burning off as it does only partially and with some difficulty, it must, in order to hold melted metal, be held firmly in the crucible shape given to it and this is effected by clay, that when wet mixes easily with the powdered black lead, and when dried and burned holds it rigidly and at the same time resists the highest white heat of the furnace. A clay from Klingenburg in the Palatinate seems to be pre-eminently adapted for such crucibles, and I strongly advise employing exclusively the best quality, every lump of which has the Government stamp impressed on it.

I have tried lower grades of the Klingenburg, and some of our own clays (Amboy, &c.), but I have found none to combine the excellent qualities of the best Klingenburg. In its wet state it is superlatively plastic, and free from every trace of grit, and in this state can be blended most intimately with the fine scales of black lead. It is barely fusible by itself at a white heat, and

when mixed with an equal quantity of graphitic scales, as in the crucible mass, it only softens at a white heat so far as to surround each scale, which it holds immovably in its place, while the equal quantity of scales prevents any further fusion, even when kept for 10 or more hours at the highest white heat. Viewed chemically, Klingenburg clay is a hydrated silicate, containing in round numbers about $\frac{3}{8}$ alumina, $\frac{1}{8}$ water, and the balance silica, with minute quantities of oxide of iron, and alkaline fluxing matter. In its employment for fire crucibles, the only consideration is its content of $\frac{1}{8}$ water when calculating the weights of the ingredients.

Sand.—The third constituent of the graphite crucible is a rather coarse siliceous sand that will stand a high heat without flying into fine powder. Its action is mechanical, causing a more uniform air drying, previous to burning, so as to avoid cracking, and playing the limited part of a skeleton to maintain the shape of the crucible. A good infusible clay, hardened by burning, and broken and sifted as a coarse sand, was found to act as well as sand, but not perceptibly better.

Mixture for Crucibles.—The only point of agreement in the composition of the best crucibles seems to be that when burned they should contain over 50 pr. ct. carbon, as determined by analysis. The formula may vary within rather wide limits, but our experience with good crucibles of the best makers and with our own, indicated that the burned crucible should show 55 pr. ct. carbon on analysis. In calculating the quantity of black lead used in a crucible from the determination of carbon, it is only safe to add 6 pr. ct. for the siliceous sand and pyrites found in the black lead. Some of the choicest black lead grains now in commerce have scarcely 1 pr. ct. of foreign matter, so entirely has the earthly constituent of plants or the small percentage of ash in anthracite disappeared from the mineral graphite. The proportions of black lead, air dried clay and sand used by different makers, and by the same maker at different times, vary considerably, but the following is given as illustrating approximately the composition for making the convenient amount of about 2,000 lbs. of crucible mixture when burned. As a crucible maker, I prefer the weight of black lead to be 1,100 lbs., so that the burned clay and sand

may together make up about 1,000 lbs., which will give the 55 pr. ct. carbon in the burned crucible:

Black Lead.	Air Dry Clay.	Sand.	Total, when burned.
1,100	1,000	120	2,100
1,100	950	160	1,996
1,100	900	200	1,990

Every manufacturer prefers such composition as, in his judgment, works best in practice.

Mixing.—The air-dried clay of a given weight for a batch of crucibles, after being roughly broken by hammer, is covered with water, and after being softened, by standing, to a thin paste, receives the due proportions of the finely ground and sifted black lead and sand, and is then partially incorporated by a shovel. Some makers, ambitious of reputation for quality, judiciously pass their hands through the incorporated mixture to detect and remove lumps of gravel of pyrites or quartz. No amount of hand or shovel mixing can equal or supersede the operations of the Mixer or Dolly-tub, a wooden vat of 4 or 5 ft. diameter and height, standing on end, to which the somewhat stiff pasty mass is transferred. Its simple machinery is a vertical shaft, revolving on the centre of the bottom, and provided with a series of horizontal knives, arranged at a distance from each other spirally, and extending nearly to the sides of the tub. The revolving knives, having their blades at a slight angle to the plane of revolution, cut the putty-like mass into separate strips which fall over the sloping blades, and again unite in neat layers, to be again cut, separated, reunited, in new positions. After thus working for some time a slide on the outside of the tub, near the bottom, is drawn and the blades slope in such a direction to their revolution that they pass the mass downwards and out of the sliding door. The protruding masses are cut off and thrown in above, and this operation, often repeated, has the effect of blending the whole into a stiff paste of absolutely uniform composition, so that each flake of the graphite, $\frac{1}{1000}$ inch diameter, is enveloped in plastic clay, and every particle of clay is in contact with a flake. The wonderful strength of the black lead crucible is due to this microscopic blending of graphite and clay.

To make the crucible still more perfect the finished mass of about a ton in weight should be laid by in a damp cellar, covered with cloths, and occasionally moistened. In spite of the restless and reckless driving of our American manufacturers, they recognize that in all clay wares, the ready-mixed masses kept for a long time make better wares than those freshly squeezed out of the Dolly tub. The experience of the Chinese for ages in making the finest clay wares, led a potter to accumulate a quantity of mixed batches, ready for use, during ten, twenty-five and more years, to be used by his son, grandson, etc., as in the case of some that had been similarly bequeathed to him.

Can this part of the manufacture of graphite crucibles, *i. e.*, making the dough, be improved? I offer the two following suggestions for consideration: 1. Scarcely any native clay is wholly free from occasional lumps of iron pyrites, which, happening to become located in the side of a crucible, might ruin the metal near it, or make a hole for the metal to escape, and in either case injure the reputation of the maker. Careful makers sometimes feel for such in the soft clay mass, but why may not this mass be passed between rollers of very small diameter, which would not pass the small gravel or lumps, but accumulate them on the feeding plate? If such arrangement will pay in brick or terra cotta ware, surely the costly crucible mass would bear it. 2. The roughly mixed crucible mass, made very liquid with water, might be passed through rotating sieves, of smaller meshes than the sand, and the slip evaporated to proper consistence on shallow furnaces similar to those employed in drying white lead. These suggestions can only be answered by the crucible maker on the ground of perfection of work, and economy.

Moulding.—All graphite crucibles, between Nos. 30 and 80, embracing those for steel, brass, silver, etc., are formed on plaster molds, centered on the potter's table, or throwing lathe. The weighed mass for a crucible, being repeatedly cut and "slapped," is pressed into the bottom of the mold, which shapes the outside of the crucible, and while rapidly revolving, a steel or iron profile of the interior is gradually lowered into the mass, which is thus pressed against the sides and raised to the top of the mold by revolution, and gives the form of the interior of the crucible. This re-

volving method, like the ancient potter's throwing lathe, is far more important in this art, because it effectually disposes every scale of graphite, tangentially to the cylindrical or conical walls of the crucible, not merely on the inside and outside, but throughout the whole mass. Here lies the great strength of the graphite crucible. It is a mass of strong scales of carbon, that will not break at right angles to their lamination, being rigidly held in their sheet-like disposal by burnt clay, that will not yield to any heat. It is a graphite crucible, infusible and unalterable, where the black lead is very much, and for a long time, prevented from burning away by its envelope of clay. It is a clay crucible, which will not soften in the highest ten-hour, white heat employed in the arts, and will endure ten times the roughest usage of any other material, except bar iron. For many years I employed excellent wrought iron crucibles for melting silver, but find that black lead crucibles are better adapted to the purpose.

The proposition to make a cheaper graphite crucible by merely pressing out the soft crucible mass in molds, without the loss of time in revolving, would utterly fail for lack of the strength of structure given by revolution. It like manner the suggested substitution of anthracite, or any other form of carbon, for graphite, would signally fail to approximate to the strength and other qualities of a black lead crucible.

Drying is effected by keeping the formed crucibles in the plaster molds for a time, and then ranging them on shelves in warm, dry air, most economically in the spaces around the upper half of the burning kiln. If dried too rapidly, or if frost be allowed to enter the drying-room, fine cracks, like crooked hair lines, barely visible, around the interior, indicate that the crucible should be remade from the foundation.

Burning.—The crucibles must be thoroughly burned, and yet so as to avoid burning out black lead, their chief source of strength and value. To attain this object they are burned in seggars, which more or less exclude air, using two seggars for each crucible, one inverted over the other, and yet graphite is sometimes partly burned out by the air entering between them. This has been partially obviated, by some makers, by smearing the joint on the outside with clay slip. Another method which I invented,

but never put into practice, as I soon after gave up the manufacture, consisted in replacing the oxygen of the air in the crucible and seggar with carbonic acid. I proposed to effect this by putting a small quantity of anthracite, or of rather dry bitumous coal, around the inner part of the lower seggar, and also, if practicable, a little of the same inside the crucible itself, avoiding defacing scratches or marks, which would be suspicious in the eyes of a buyer. I still suggest this idea to the manufacturer, for as long as there is present an easier burning carbon than graphite, the former will be attacked to the safety of the latter; and, moreover, the carbonic acid formed and forming from the coal, excludes the oxygen of the air, or the latter is consumed immediately on entering. A dry gaseous coal that neither puffs nor melts would be best for this purpose.

To secure equal and uniform distribution of heat, the furnace should have, and usually has, many openings as firing hearths around its base, and when the burning is nearly complete, these should be more or less closed, so as to prevent the entrance of more air than is barely necessary to maintain the diminishing combustion while the furnace is beginning to cool. This is another method of preventing the loss of black lead, and, with the same object, the entrance of air from above downwards should be prevented or counteracted.

The amount of carelessness or ignorance, or lack of attention to prevent burning out black lead and possibly impairing the strength of a crucible, may be readily ascertained on the burned ware. When the outside of a crucible has the usual black lead color, it is either not burned enough, and then quite soft to the nail or knife, or it is extremely well burned, to be determined also by hardness under knife or nail, or it may have received the outside cleanly operation of a brush, which is, of course, only outside, and condemnatory. An outside drab color, which is the prevailing one, indicates burning off black lead. If the drab color be very shallow and show the subjacent black by friction of the finger, then it is well burned, like the greater part of the crucibles in commerce. Even if a slight scratch with a knife show the black immediately under the outside, it still shows excellent burning. But if it require a little digging into the drab before coming to the black lead,

say ¼ inch, I should be disposed to throw off one-fourth, or more, of its value. If it go to a half inch, I should reject such a crucible as worthless, because a large proportion of its life, its strength, has been burned out.

Use and Life of Black Lead Crucibles.—The most extensive use of black lead crucibles is to melt blistered steel and cast it into any desired form, whence it is termed cast-steel. Tens of thousands of crucibles are weekly employed for this purpose, the amount of steel at each melt being 75 to 90 pounds, and a number of such melts constituting a day's work. Hence the great multiplication of furnaces, or of crucibles in a gas furnace, when 1,000 pounds or more are cast at a time. The life of a crucible for melting steel varies with the nature of the steel to be cast. If it be a highly carbonized (fine) steel, a moderate white heat will soon melt it, and the crucible may be repeatedly used. If a low (common) steel be employed, approximating to bar-iron, such as a locomotive driving wheel, the crucible has to endure five (5) and more hours of the most intense white heat for a single casting, and yet it can endure several such meltings. A crucible will bear repeated meltings of tool steel, and in order to extend its life, the outside is often coated with a good clay slip, with or without an admixture of black lead, after each melt. The wonderful protective power of the clay, embracing each particle of black lead, shows the advantage of grinding the black lead to great fineness, making clay and graphite mutually protective. In melting steel, the melter has to consider the length of the time of melting, and, therefore, the quantity of additional carbon the steel is assuming from the black lead pot, which excess he neutralizes by adding a certain quantity of scraps of bar-iron to the melt.

To show other possible applications of the crucible mass I may mention that syphons have been constructed of it, and successfully employed for transferring melted cast-iron from one vesssl to another.

Black lead crucibles are also extensively employed in melting brass, bronze, German silver, gold and silver. I here add a few data in relation to the last two metals. The crucibles employed at the U. S. Mint, at Philadelphia, are so called No. 70 pots, of three gallons capacity—being 9 inches in diameter at the top and 13

inches deep, both inside measurement. The walls are 1 inch thick at top, and 1½—2 inches at bottom. In such a crucible we usually melt at once, every 1½ hours, 5,400 ounces of standard gold ($\frac{9}{10}$ pure), or 370 pounds, avoirdupois; four melts, a moderate day's work, equal, therefore, 1,480 pounds, or 21,600 ounces. The value of such a single melt is over $100,000. One such crucible is run for four days with full confidence in its safety, and fearing no fracture or loss of gold; for although we have run them for six (6) days it is found the economy does not balance the possible danger of fracture and leaking. Taking the safe run of four days, we melt in a single crucible, with scarcely any loss, $1,600,000 worth of gold at the crucible cost of about $2.50.

We usually melt 3,500 ounces (=240 pounds, avoirdupois,) of standard silver in a crucible (No. 70) at one melt, and cast six such melts, without undue haste, in a day, making 1,440 pounds, or 21,000 ounces. Since it is safe to estimate its life at five days, we melt in one such crucible before it is laid aside 7,200 pounds (=105,000 ounces) of standard silver, or about the same amount in dollar value. This, however, is not all of its life, but only its manhood, for after doing duty for so long a time in perfect health, it is put aside to purify and toughen inferior silver and to gather into melts grains and residues, otherwise wandering off, for some time before it is thoroughly disabled and ground to powder, to recover the precious grains which it has concealed in its body.

To one who knows the ease with which alkalies attack clay at a high heat, and how readily fusing nitre will burn off carbon, even black lead, it is quite a surprise to witness the wonderful durability of the black lead crucible, when in its nearly worn-out condition it is used for fluxing residues, and we can only attribute its strength to the minute and intimate blending of clay and graphite, mutually resisting destruction.

No doubt the life of these crucibles is prolonged in melting bronze, brass, &c., because they are so tough through the blending of clay and graphite that they rarely break suddenly, but rather give notice of their intention to do so by gradually opening, and even then not until they have been worn thin by long use.

Some years ago I prepared some black lead crucibles with more than usual care in order to test their life. We made from 40 melts

of silver, the lowest limit, to 60 melts, the higher limit, in a single crucible. I have no doubt that a careful study of the principles of the manufacture of black lead crucibles here developed, somewhat fully, and even perchance their further development, beyond my conception, can improve their quality so as to double the life and strength ascribed to them.

U. S. MINT, PHILADELPHIA, Dec. 31, 1884.

NOTE ON CAMPHOR MOTIONS.

By P. CASAMAJOR.

On the 4th of October, 1877, I read a paper "On the Motions of Camphor on the Surface of Water," before the American Chemical Society, in which I described experiments, which had led me to the conclusion that these motions were due to electricity.

The extraordinary motions which give an appearance of life to pieces of camphor, swimming on the surface of water, are not to be seen at all times. Very often camphor will remain motionless, while at other times the pieces gyrate with great animation. One of the earliest observers of these singular motions, Romien (1748), came to the conclusion that they were due to electricity, while subsequent investigators, among whom may be counted the great Volta, have generally decided that there is no connection between electricity and the motion of camphor on water.

I was led to believe that camphor motions were due to electricity by the results of experiments, of which I will give a brief account.

When pieces of camphor are thrown on water, they may remain torpid or they may gyrate with every appearance of life. In the latter case, the motions may be instantly arrested by dipping a finger in the water on which the camphor moves. If we have pieces of camphor lying quietly on water, they may be made to move by dipping into the water a rod of either glass, sealing wax or vulcanite, electrified by friction. After every immersion the glass is to be dried by wiping with a dry cloth or a piece of bibulous paper; and, before every immersion, the rod is electrified by rubbing with a piece of silk or flannel. After one or more immersions of the electrified rod, the camphor motions invariably start, and by a few additional immersions they increase in intensity. The fact that these

motions could be started by means of an electrified rod was a new one. Those who had decided that camphor motions were not due to electricity were not acquainted with it, and I was led to believe by this fact, and by others which I gave at the time, that camphor motions were electrical phenomena. There was, however, something unsatisfactory in this method of applying electricity to the water on which pieces of camphor floated, but, at that time, no other method occurred to me. Afterwards the whole subject slipped from my mind, but, quite recently, the following experiment has suggested itself, which seems to show that camphor motions are not due to the state of electrical tension of the liquid on which pieces of camphor float.

The experiment was performed in a glass dish about two inches deep, and of five inches diameter. This was filled with water up to within an inch of the top. The bottom of the dish and the sides, up to within an inch of the top, were covered with tin foil. A metallic wire dipped in the water of the dish, with one end out of the water, but having no connection with the outside lining of tin foil.

This glass dish was the equivalent of a Leyden jar, which could be charged with electricity from the plate of an electrophorus by touching the wire dipping into the water with this plate, laying a finger at the same time on the outside armature.

A few pieces of camphor were placed upon the water in the dish, and these were reduced to immobility by dipping a finger in the water. After this the water in the dish was charged with electricity by repeatedly placing the plate of an electrophorus in contact with the wire dipping into the water, while the outside armature was touched with a finger.

There was no effect produced on the pieces of camphor floating on the water of the dish. These remained motionless, not exhibiting the slightest tremor. There could be no doubt that the water surface was in a greater state of electrical tension in this experiment than when the water is electrified by dipping an excited rod in it, for, by placing one hand on the outside armature of the dish, and slowly approaching the water surface with a finger of the other hand, a perceptible spark was obtained.

There seems to be a discrepancy between the results to be obtained in these two classes of experiments.

By dipping an electrified rod successively in water the camphor motions always take place, while, when we electrify by applying the plate of an electrophorus to the water in the dish, as described, not the least tremor can be perceived. As these results were obtained over and over again, there cannot be any doubt as to the difference of the effects in the two modes of operating.

The conclusion that may be drawn is that camphor motions are not caused by electricity. In the case of an electrified rod, dipped successively in water, I am inclined to the opinion that electricity acts indirectly by removing physical impurities from the surface of the water, as shown by the following experiment :

Take a glass of water, and on its surface place a few pieces of camphor. If these are inclined to move, they may be stopped by dipping a finger in the water. Then blow a cloud of lycopodium powder over the surface of the water, so as to cover this surface uniformly with the powder. If now an electrified rod is dipped repeatedly in the water, wiping it dry after every immersion, and, rubbing it with a piece of flannel before dipping in the water again, the result will be that the pieces of camphor will move as if gifted with life. It will be noticed at the same time that the surface of the water will be almost entirely free from lycopodium, as the powder has been gradually removed by each successive immersion of the electrified rod, which attracts lycopodium like all other light bodies. If now we suppose that films of oily matter are removed in the same way by an electrified rod, we may conceive how electricity can act indirectly in removing impurities from the surface of the water.

There would remain to explain why films of oily matter or other impurities act as a check on camphor motions while a physically clean water surface is a favorable condition in producing these motions. Towards the solution of these questions I will venture the following explanation, which may serve as the base of further researches.

It is a well ascertained fact relating to camphor motions, that when these motions take place the camphor dissolves in water more quickly than when the camphor is at rest. I think that a connection exists between camphor motions and the three following facts :

1st, that camphor dissolves in water quite easily at times ; 2d,

that its density allows it to float on water ; and 3d, that the solution has a density slightly different from that of water. I am not prepared at present to develop these ideas, which would require further experiments to establish.

I will confine myself to the well ascertained fact that a piece of camphor wears away much faster when in motion than when at rest. This being established, it appears more than probable that if a piece of camphor is perfectly free from oily matter it will dissolve in water more readily than if protected by a greasy film. The slightest film of this nature, in contact with camphor, becomes a saturated oily solution of camphor, and if any excess exists, over what will cover the camphor, the greasy film will extend over the surface of the water.

When things are in this condition, if an electrified rod is dipped several times in the water, every immersion will remove a portion of oily film from the surface, until finally the film on each piece of camphor becomes so thin that the water reaches the pieces of camphor, and these immediately become gifted with motion.

ON THE METHODS OF INDIGO TESTING.

By Henry M. Rau, Ph. D.

It appears strange, when the high price of indigo and its large consumption in the industrial arts are considered, that the methods commonly employed in this country for its valuation should, from a scientific standpoint, be so crude and inaccurate.

Taking the average price of the various grades of indigo in the market as a basis for calculation, it may be stated that a single per cent. of indigotine represents, to the consumer, from two to two and one-half cents for each pound of goods purchased.

Under these circumstances it would seem highly desirable to employ tests as accurate as possible, even though these should not be as time-saving as the greater number of commercial tests.

Nevertheless, it is a fact, that the ordinary indigo "analyses" are so wide of reliable results, that guess work might quite as well be substituted for them, and this in face of the fact that we have in the gravimetric determination by the reduction methods, a means for a closer valuation of indigo than can be applied in the case of almost any other dyestuff.

The ordinary mode of judging the quality of an indigo and by which it is commonly bought and sold, is quite superficial; and even in experienced hands allows but a rude estimate of its value. The buyer or broker takes a cake of indigo, breaks it with a knife, and compares it with another of previously known worth as to color, density, hardness, etc. Upon these points of comparison he relies for a correct valuation.

Where a formal analysis is required, the oxidation tests are usually employed; and it is to the inadequacy of these that I particularly desire to call attention. They are based upon the oxidation of the sulpho acid of indigotine in aqueous solution by a variety of agents, with the destruction of the blue color. The different processes in use employ potassium permanganate, potassium bichromate, bleaching powder, or nitric acid, as the case may be. The sample of indigo is dissolved in strong sulphuric acid and the solution brought to a certain volume by the addition of water. A measured quantity is then decolorized with an oxidizing solution which has been standardized upon pure indigotine or some indigo of known value. When the blue color of the indigotine solution has been destroyed and been replaced by a yellow or ochreous shade, the oxidation is complete and the volume of the reagent solution employed, is noted. Now, if indigo contained merely the coloring principle, indigotine, in mixture with inorganic matters, this volumetric estimation would be both practical and reliable, but as its various other ingredients (which are entirely valueless) also enter into the reaction and consume a quantity of the reagent, the method gives these useless matters the same value as the indigotine. The figures obtained are, therefore, always too high, and that in no uniform ratio, but varying with the amount and character of the accompanying impurities. The color which the latter themselves impart to the solution also renders it very difficult to determine exactly when the requisite quantity of the re-agent has been added.

The reason why these methods, although known to be so faulty, have been retained in general practice, is to be found in the high figures obtained by them, which are to the apparent advantage of the dealer. Their scientific appearance also is calculated to impress the unskilled. They are, in reality, anything but scientific. A good Bengal indigo which actually contained 52.5% indigotine, showed

72.5% when tested by the permangate process, a Kurpah of 41.8% (with a larger proportion of organic impurities) by the same test yielded 69.2%. Using potassium bichromate as the oxidizing agent, the figures obtained were nearly the same. In fact, in order to achieve correct results by any of these methods, it would be necessary to determine previously the precise amount of extraneous organic matter accompanying the coloring principle.

I have recently adopted for the analysis of indigo a very convenient modification of Fritsche's reduction test, which is also sufficiently rapid to permit as many as three determinations to be made together within twenty-four hours. I proceed as follows: From 1½ to 2 grammes of the sample, in very fine powder, are carefully weighed and placed in an 8 oz. Erlenmeyer flask (A). The flask is provided with a doubly perforated rubber stopper, through which passes a bent glass tube (B) provided with a stop-cock and reaching just below the stopper, and a second tube (C) of syphon shape which passes nearly to the bottom of the flask and terminates in a small funnel.

In this funnel is placed a wad of glass-wool. Tubes and stoppers must fit air-tight into the flask. The apparatus is accurately tared; from three to four grammes of pure grape sugar in small pieces are then placed in the flask, fifteen to twenty c. c. of a 40%. caustic soda solution, sixty c. c. of water, and sufficient 90%. alcohol (120 c. c.) to bring the whole to about seven fluid ounces, are added. The apparatus is then weighed a second time, so that by deducting the tare of the flask, etc., we obtain the weight of the total contents. A small piece of rubber tubing closed with a pinch cork, is slipped over the syphon tube, the stop-cock is closed, and the flask heated on a water bath for twenty-five to thirty minutes. At short intervals the stop-cock is quickly opened and

closed, to relieve the pressure of the alcohol vapors, the flask being occasionally shaken. The indigo readily dissolves, the liquid assuming a deep yellow color, the insoluble substances settling clearly to the bottom. The solution being completed, the flask is allowed to stand for about an hour, and is then connected with a generator of carbon dioxide gas.

The clear liquid is quickly run off, as far as possible, through the syphon tube, the glass wad retaining any floating particles, and the flask is again weighed quickly, whereby the weight of the liquid thus run off is ascertained. This portion is placed in a beaker glass and a stream of carbon dioxide gas passed through the same for fifteen minutes, causing the indigotine and indirubine to precipitate in crystalline flakes. A current of air is then drawn through, which completes the precipitation. The precipitate, which by this means is obtained in beautiful copper-colored flaky crystals, is collected upon a previously dried and weighed filter, washed repeatedly with boiling water, then with hot dilute hydrochloric acid and finally, with water. Precipitate and filter are then dried at 110° C., and lastly weighed. Deducting the weight of the filter, the indigotine and indirubine contained in that portion of the liquid treated as described, are obtained, and by a simple calculation the percentage contained in the whole sample is ascertained.

The advantages of this process are found in the fact, that the insoluble impurities in the indigo are readily deposited, thereby avoiding the tedious filtration of the liquor, and that the indigotine and indirubine are obtained in a crystalline precipitate which is easily purified by washing.

Schunk has stated (Lit. Phil. Soc., Man. XIV.) that where he employed small quantities of indigo, as compared to the quantities of alcohol, caustic soda and grape sugar, Fritsche's process occasionally yielded him very incorrect results, the indigotine at times not precipitating at all. However this may be, the results which I have obtained by this method have been surprisingly uniform. Thus a sample of artificial indigotine, prepared from propiolic acid, tested 99.96%.; two analyses of pure dry indigotine from indigo showed 99.68%. and 99.74%.; three estimates of a high type Bengal indigo gave 60.35%., 60.78%. and 60.72%.

Few analytical methods can claim greater uniformity than this.

A NEW FORM OF DROPPING FLASK.

By F. V. Pool.

It is often convenient in methods of titration in the laboratory to make use of a flask or bottle from which the standard solution can be poured or dropped and the amount of solution which has been used, determined by weighing the flask or bottle of liquid before and after the operation. This method has several advantages over the ordinary volumetric one in which a burette is used, the first advantage being that of using the liquid at any ordinary temperature without making a correction for the difference between that temperature and the one at which the liquid was standardized, provided, of course, that the standardizing was made with the flask and not with a burette.

This is due to the simple fact that a gramme of water or any other liquid *weighs* the same, no matter whether the temperature be 15° C. or 20° C., and if, in process of standardizing, for example, a solution of $AgNO_3$, it is found that one gramme of the solution is equivalent to one milligramme (.001 grm.) of Cl, we may use the solution (by weight) afterwards at the same temperature or any other temperature ordinarily found in the laboratory without making any correction for the same. This is not the case in using an ordinary burette and reading off the *volume* taken, for here we must make use of a table of corrections for temperature, which would give us for a range of 10° C.—say, between 25° C. and 15° C., a difference of .2 of 1% of the strength. That is to say, if 100 c.c. of the said solution are equivalent to .100 grm. of Cl, at 25° C. the same *volume* of the liquid at 15° C. would be equivalent to .1002 Cl. Of course this applies to all burettes which record the volume of liquid taken.

In the second place the dropping flask has an advantage over burettes which require the use of a pinchcock and rubber tube, inasmuch as we may employ in the flask a solution such as permanganate of potash or chromic acid which would act injuriously upon the rubber tube and become itself decomposed. To obviate this difficulty in the use of burettes, recourse is had to glass stopcocks, which are expensive and not always perfect. They are also liable to stick very tightly if allowed to remain for some time in contact with K HO or Na HO solution.

The Gay Lussac and Bink burettes are not open to the second

objection, but they are attended with a difficulty which is peculiarly their own, namely, the retention of a drop of the solution at the end of the dropping tube by capillary attraction and the prevention of the free delivery of the contents of the burette.

Analysts have overcome this difficulty by inserting a stopper, provided with a glass tube and rubber mouth tube, into the top of the burette and expelling the refractory drop with the breath. This procedure answers very well, but there is a somewhat neater way as we shall see further on. The dropping flasks in use at the present day are few in kind and very simple, being nothing but small flasks of varying capacity provided with a top or side tube and open to the objection which is found in using the burettes of Gay Lussac and Bink. The flask here shown is an improvement upon existing forms, and has been used for some time in the laboratory and with a great deal of satisfaction.

It consists of a light, flat-bottomed flask of a size determined by the capacity of the balance upon which it is to be weighed. From one side, near the bottom, a narrow tube starts and reaches up a little higher than the body of the flask, being then turned outward like the side tube of Gay Lussac's burette, which it resembles. In the neck of the flask a small hole is blown which, during the operation of dropping, is governed by the thumb of the manipulator. Into the neck of the flask a rubber stopper is fitted and through this stopper a short thistle-tube is passed, the upper part of the latter being covered by a piece of thin sheet rubber, securely fastened by a piece of thread. The flask being filled with the standard solution is first counterpoised on the balance. It is then grasped around the neck, the thumb being placed very near the small hole in the side and the forefinger held just over the rubber membrane on the top.

The flask is then tipped so as to deliver the solution through the side tube, the rapidity of flow being governed by the thumb, which closes the small hole in the neck at will. If at any time during the operation a drop is formed at the end of the tube, it can easily be forced out or drawn back into the flask by the control

of the thumb and forefinger. By this means any amount of delivery may be obtained from part of a drop (by touching the end of the tube to the stirring rod) to a steady stream.

After a sufficient quantity of the solution has been used, its weight is determined by replacing the flask upon the balance pan and adding weights to balance the counterpoise in the other pan. In our own laboratory we use for this purpose a balance whose capacity is 500 grammes in each pan, and a counterpoise consisting of a small pasteboard box partly filled with shot. With this it is the work of only a few seconds to counterbalance the flask, and after the titration is completed, the amount of solution used is quickly ascertained by the above method of leaving the counterpoise as it was and adding weights to the pan containing the flask. The capacity of the latter is about 250 c.c. and one filling suffices for several operations, the counterpoise being changed each time by removing some of the shot.

The convenience of this modified volumetric method can be appreciated by those who usually have a number of determinations of one kind to make in a day, and it will recommend itself to all on account of the ease of manipulation, accuracy, and slight cost of the apparatus.

The adaptation of the above described contrivance to a Gay Lussac burette, is obvious. All that is necessary is to adapt a rubber stopper and thistle-tube to the burette, and to blow or drill a small hole into the side of the burette about an inch from the top.

The flasks used were made by William Baetz, 96 Fulton Street, New York.

NOTE ON OIL OF MALT.

By Narcisse Pigeon. (Abstract.)

Beer is properly manufactured from malt and hops, barley being the grain usually employed in making malt. None of the substitutes for barley malt, contain the characteristic oil of malt and lack therefore the proper taste and aroma. Oil of malt has been found by the author in the radicles or rootlets of barley malt, and he suggests its extraction as a proper substance for developing the taste which is lacking in beer made from the above-mentioned substances.

ABSTRACTS.

GENERAL AND INORGANIC CHEMISTRY.

On Nitrification.—Part III. R. Warrington.

A summary of results obtained at Rothamstead during the last few years in continuance of previous work. Nitrification is produced by an organism which the author now believes to be a bacterium. The evidence that an organism is the agent in nitrification is now so strong, that the burden of proof is thrown upon those who deny the statement. The process may be started at will in any suitable solution by addition of a nitrified material, while such solutions, unseeded, remain sterile for years. Phosphoric acid is essential to the continuance of the process as it is to the existence of life. The range of temperature within which nitrification is possible, is the same as that which limits the vitality of low organisms, and the process requires presence of the plant food suited to such organisms. Antiseptics are fatal to nitrification; a temperature of 100° C. is also fatal. The process may be permanently arrested in sewage, by boiling, if introduction of foreign matter be subsequently prevented, but is started at will, after boiling, by the introduction of a nitrified solution. Finally, the theory that nitrification in the soil is due to condensation of gases in the pores of a soil by which an exceptionally active oxidation is brought about, is overthrown by Schloesing's experiments, which show that such gases occupy normal volumes.

All of the Rothamstead experiments of this series were made with solutions. Those most commonly employed contained ammonium chloride, 80; sodium potassiums tartrate, 80; potassium phosphate, 40; and magnesium sulphate, 20; the numbers representing mgms. per litre. Calcium carbonate or gypsum, or in some cases, tricalcium phosphate, was added. Diluted urine, (4 c.c. per litre) was also used. All the solutions were sterilized by heating to near 100° C. for 6–8 hours, and then seeded with natural soil or a nitrified solution.

The nitrifying power of the soil at different depths was determined by taking samples of earth, with proper precautions, from the side of an excavation in a cultivated field, at stated depths from

of the thumb and forefinger. By this means any amount of delivery may be obtained from part of a drop (by touching the end of the tube to the stirring rod) to a steady stream.

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The nitrifying power of the soil at different depths was determined by taking samples of earth, with proper precautions, from the side of an excavation in a cultivated field, at stated depths from

the surface, down to nine feet. The organisms in question are shown to exist mainly within nine inches of the surface in a clay soil, rarely extending below 18 inches, and never, so far as these experiments show, below three feet.

All nitrogenous substances which yield ammonia by the action of organisms in the soil, are nitrifiable. Nitrification is limited by excessive alkalinity of the medium as has been shown by others. Under given conditions, an alkalinity equal to 160–200 mgms. of disodium carbonate per litre was favorable to the process, while 320 mgms. or more proved unfavorable. Strong urine is, therefore, unfit for nitrification, a fact which has an important bearing upon the use of liquid manures. Apart from alkalinity, also, concentration of ammoniacal solutions has an influence upon nitrification.

The rate at which nitrification proceeds depends also upon the vigor of the organism, which, in turn, rests upon the conditions of nutrition, access of oxygen, etc., in its previous development and in its ancestry. Depth of solution, connected as it is with access of oxygen, has an important influence upon nitrification. In porous soil the process is much more favored in respect to oxygen than it can possibly be in solutions. Organic carbon is essential only as it is required for the nourishment of the organism, and excess is rather prejudicial, because it tends to appropriate a part of the necessary oxygen. Urine contains sufficient organic carbon for its own nitrification. Nitrification may go on slowly at temperatures as low as 3.2° C.

When a fresh sample of nitrified soil is added to a nitrifying solution, the first effect is a reduction of nitrates, already present, to ammonia. This reduction runs its course more quickly than the subsequent nitrification; it is accompanied by turbidity of the solution, and has been shown by Dehérain and Maquenne to be caused by bacteria.

As to the theory of oxidation of ammonia to nitric acid in the human body, the author has made direct experiments, in which the absolute quantity of nitric nitrogen eliminated in equal times was determined, while stated quantities of ammonia salts were taken into the system. The results lend no support to a belief in such oxidation of ammonia. A. A. B.

On Germination in a Soil Rich in Organic Matters, but Free from Microbes. E. DUCLAUX.

The destruction of the organic matter of soils by microbes, and the production on such soils of new vegetation are phenomena which always occur together. Hence the inquiry whether plants can develop in the absence of microscopical organisms. In other words, could the plant, without such organisms, utilize the organic matters left in the soil by preceding plants? This problem depends on this other problem. If a seed is sown in a soil rich in organic matter, but free from microbes, can it utilize manure placed in this soil?

Among substances which can be offered to a seed as manure, the author has chosen milk, which contains a carbohydrate, milk sugar, and a nitrogenous substance, caseine. The author has previously shown that milk is only assimilated by animals after treatment by two ferments—rennet and casease.

The author operated on two seeds, peas and beans. These were freed from microscopical organisms and planted in a sterilized soil moistened with sterilized milk.

At the end of two months, the milk remains unchanged—it has not been coagulated and its caseine can be precipitated by acids.

With solutions of rock candy and with starch paste, analogous results have been obtained. The plants produced from seeds in such conditions are exactly as if grown in pure distilled water. The weight of each plant is less than that of the seed from which it grew.

In presenting the above paper, M. Pasteur made the following remarks:

"Often in our laboratory conversations, I have called the attention of young scientists to the interesting results which would be obtained by feeding a young animal, from its birth, with substances pure from microscopical organisms. If I undertook this study it would be with the expectation that life, under such conditions, would not be possible. If such experiments were undertaken it would be well to try the influence on digestion of the addition of any particular microbe or of an association of known microbes of various kinds. Hen's eggs would afford a very convenient agent for such experiments. The egg should be deprived of all living

dust, at the moment when the chick is to appear. This should be placed in a space free from germs. The air should be renewed in this space, but all the air introduced should be freed from germs. All the food introduced should also be freed from germs (water, milk, seed.) The result of this experiment may either confirm my preconceived ideas, or it may show that life is not prevented, or that it takes place even more actively than usual. At any rate, the result obtained would be interesting." (*Comptes Rend.* **103**, *66.*)

P. C.

An Explanation of Gladstone and Tribe's "2–3 Law in Chemical Dynamics." J. W. Langley.

"In a paper by Gladstone and Tribe on "A Law in Chemical Dynamics" (*Jour. Chem. Soc. 1871, p. 1123*), it is stated that, if a plate of metal be suspended in a solution of a salt of another metal which it precipitates, the percentage of salt in solution will be to the rate of chemical action as the powers of 2 to the corresponding powers of 3. This is the "2–3 law" referred to, and it has been found to hold good for solutions of 3 per cent. or less. No explanation of it has been given, and the author of the present paper has sought to distinguish between the chemical and physical elements of the problem, and to define the value of each. To test the influence of the products of chemical action upon the rate and continuance of that action the suspended plate (of copper, in a solution of silver nitrate) was slightly curved and made to revolve in the solution so as to present fresh portions continually to the action of the plate and thus to give the effect of more rapid removal of the solution of copper than was effected by gravitation in the case of the stationary plate. The experiment was varied also by the use of a revolving brush which swept one surface of the stationary plate (the other surface being varnished) at a uniform speed, and by revolving the precipitating vessel itself in a vertical plane at such a rate that the tendency to remove the products of chemical action from the plate was 30 times that of gravity. The results show that the rate of action is dependent both upon the strength of the salt solution and upon the rate at which the products of chemical action are removed from the surface of the plate. As the stronger solution produces more rapid action it yields also a denser product, which

falls away more rapidly, and, for given conditions, within the limits of the original experiment, the law can be shown to be a necessary result. That this is true only for conditions in which gravity effects the removal of the products of chemical action from the plate, is demonstrated by the departure from the law in the case of the centrifugal experiment. The whole question is one relating to the influence of mass upon chemical action. The author concludes that the true law of chemical action in such cases as this "should be that the time during which one atom replaces another is constant and that the total chemical action varies directly as the mass of the reacting body in solution.—(*Jour. Chem. Soc.*, **265**, *633*).

A. A. B.

The Use of Boiling Oxygen as a Means of Cooling, and the Solidification of Nitrogen. S. V. Wroblewski.

Oxygen does not solidify when brought to boiling by sudden release from pressure. It leaves, however, a crystalline precipitate upon the bottom of the containing vessel or any object plunged in it. Further research is needed to decide whether these are oxygen crystals or the impurities in the gas. This deposition of crystals interferes somewhat with its use for cooling. A second obstacle is the necessity for using very strong, closed vessels. Only the moment of violent boiling when the pressure is removed can be utilized. This is of very short duration and presents another obstacle. The temperature of boiling oxygen, measured by a thermo-electric method, was 186° C. As yet nitrogen only has been cooled in this way successfully. It solidifies and falls in flakes which consist of crystals of notable size. (*Jour. f. prakt. Chem.*, **29**, *95*.)

F. P. V.

Solubility of Solid Bodies in Water at Different Temperatures. J. L. Andreas.

The solubilities of some of the commoner solids, determined by distinguished experimentors, show decided differences. The author points out certain courses of error, as proper shaking, regulation of temperature, inaccurate thermometers, and purity of solid. In a series of experiments he seeks to avoid these errors, determining the solubility of sodium chloride, potassium sulphate and potassium nitrate. (*Jour. f. prakt. Chem.*, **29**, *456*.) F. P. V.

Modifications of Sodium Sulphate. S. U. Pickering.

From the discrepancies in existing data relating to the heat of solution of metallic sulphates, the author was led to suspect the existence of modications of the same sulphate, and the present paper is an attempt to show that anhydrous sodium sulphate exists in at least two different conditions. The molecular heat of solution for samples prepared by drying the effloresced, crystalline salt at different temperatures is approximately constant for samples dried at or below 150° C.; dried between 150° and 200° C. the salt shows a remarkable rise in heat of solution, reaching, however, at temperatures somewhat above 200°, a value which is constant up to the fusing point. The discrepancies in the results of Berthelot and Thomsen may have resulted from the use of mixtures containing different proportions of the two modifications. The heat of solution of the fused salt presents anomalies which require further investigation. (*J. Ch. Soc.*, **265**, *686.*) A. A. B.

The Origin of Calcium Thiosulphate. E. Divers.

The action of the atmosphere upon calcium polysulphides (decomposition of soda waste) involves no direct oxidation of sulphur, but a splitting up of these compounds by hydrolysis into sulphur, H_2S and lime. H_2S is then oxidized only in respect to its hydrogen, the free sulphur reacting upon calcium hydrate to produce calcium thiosulphate and hydrosulphide.

$$3\,CaH_2O_2 + 12S = CaS_2O_3 + 2CaS_5HOH.$$

By dissecting this reaction, however, it may be shown to involve a preliminary reaction in which SO_2 is set free. The union of calcium sulphite and S to produce thiosulphate is a well known reaction and as the one other method of obtaining thiosulphate (the action of SO_2 on a hydrosulphide) involves a similar reaction, the author concludes that the origin of the thiosulphate in all cases rests upon one common reaction, viz.: *the union of sulphur with calcium sulphite.* (*J. Ch. Soc.*, **265**, *696.*) A. A. B.

ORGANIC CHEMISTRY.

On Uric Acid. E. FISHER.

Uric acid methyl-ether on being treated with phosphoric chloride loses two atoms of hydrogen and two of oxygen which are replaced by two Cl atoms, thus:

$CH_3 - C_5N_4H_3O_3 + 2PCl_5 = 2HCl + 2POCl_3 + CH_3 - C_5N_4HOCl_2$.

In this chloride one hydrogen atom may be replaced by CH_3 and moreover, the two chloride atoms by H, etc. By prolonged treatment with PCl_5, the compound $CH_3—C_5N_4HOCl_2$, loses H and O again, and is converted into the tri-chloride $CH_3—C_5N_4Cl_3$. In this, as in the previous compound, the Cl atoms may be replaced by various radicals.

The author regards these compounds as derivatives of the hitherto unknown hydrogen compound $CH_3—C_5N_4H_3$, which he calls methylpurine. Among the compounds enumerated are the following: Dichloroxymethylpurine, trichlormethylpurine, oxymethylpurine, dichloroxydimethylpurine, oxydimethylpurine, ethoxychloroxydimethylpurine, dioxydimethylpurine, diethoxydimethylpurine, trioxydimethylpurine. (*Ber. d. chem. Gesell*, **328**, *1884.*) J. H. S., Jr.

On the Presence of an Isocyanide (Carbylamine) in the Lighter Portions of Commercial Benzol. E. NOELTING.

These light distillates, known technically as "heads," have been shown by different investigators to contain carbon disulphide, crotonylene, amylene, hexylene, ethylic alcohol and acetonitrile or methyl cyanide, in addition to more or less benzol. The author has noticed the odor of an isocyanide in this product and finds on examination that this odor disappears on treatment with HCl, giving place to the odor of CS_2, while the liquid, after this treatment, is found to contain ammonium chloride and a small quanity of a hydrochloride of primary amine. The proportion of isonitrile is evidently very small but the odor of these bodies is strong and characteristic, and their poisonous properties very marked. The low boiling points of the "heads" excludes all of these bodies

except methyl- and ethylisocyanide, which boil respectively at 59.6° and 78.1° C.

The application of this product ("heads") is very limited. It has been used to dissolve caoutchouc, for which purpose, however, the naphthas of 120°–150° C. are preferred. In a note, M. Werner describes a fatal case of poisoning of a workman who inhaled the vapors of this same benzine, and experiments, made jointly with the author, upon animals, pointed to the isocyanides above mentioned as the cause of death. (*Bul. Soc. Ind. Mulhouse, Oct. 1884.*) A. A. B.

Occurrence of Citric Acid in Seeds of Leguminous Plants. H. RITTHAUSEN.

The existence of citric acid, together with malic and oxalic acids, in the seed of the yellow Lupine (*Lup. luteus*) has already been shown. The author finds it also in the seed of *Vicia sativa* (vetch), *V. faba* (hog's bean), various varieties of peas, and in the white garden bean (*Phaseolus*). The powdered seeds are digested with water acidified with hydrochloric acid, the solution filtered, neutralized with an alkaline hydrate, and precipitated with lead acetate. This is suspended in water, treated with sulphuretted hydrogen, and the citric acid gotten in the usual way from the acid liquid. The white bean contains very little citric acid and peas contain less than the other legumes mentioned. (*Jour. f. prakt. Chem.*, **29**, *357.*) F. P. V.

Melitose in Cotton-Seed. H. RITTHAUSEN.

The cotton-seed cake is treated with 80 per cent. alcohol (60–70° C.), the excess of alcohol distilled off, the residue freed from fat by means of ether, the remaining coloring matter precipitated with basic lead acetate and ammonia, and the excess of lead removed by sulphuretted hydrogen. Needle-like crystals of melitose are deposited on evaporating the solution, and these may be washed and recrystallized. The crystals, which form about three per cent. of the crude material, seem, on examination, to possess all of the properties of melitose. (*Jour. f. prakt. Chem.*, **29**, *351.*)

F. P. V.

On Paranitrobenzoylacetic Acid. W. H. PERKIN, JR., and GUSTAV BELLENOT.

It has recently been shown by Baeyer that phenylpropiolic acid ether, when treated with H_2SO_4, takes up the elements of water and is converted into benzoylacetic acid ether. The authors thought it of interest to see whether, under the same conditions, paranitrophenylpropiolic acid ether, would be converted into paranitrobenzoylacetic acid ether. This was found to be the case. After some experimenting they obtained a compound which analysis showed to be paranitrobenzoylacetic acid, formed according to the reaction:

$$C_6H_4\ (NO_2)-C{=}C-COOC_2H_5 + 2H_2O{=}C_6H_4\ (NO_2)\ CO-CH_2-COOH+C_2H_5OH.$$

The ether was obtained from the above, by dissolving the free acid in absolute alcohol, and saturating with HCl. The temperature during this treatment should not exceed 10° C. (*Ber. d. chem. Gesell*, **326**, *1884.*) J. H. S., Jr.

On the Action of Aldehydes and Ammonia on Benzil. (Continued.) F. R. JAPP and S. C. HOOKER.

The reaction between salicylaldehyde or furfuraldehyde and benzil in presence of ammonia, differ from reactions of this class previously studied by the authors. The two carbon atoms of the dicarbonyl group instead of becoming doubly linked, separate in such a way that the diketone is broken into halves, which become two substituted acid radicals in the new compound. At the same time the two aldehyde residues become linked by means of the carbon atoms of the CHO groups. The salicylaldehyde compound has been especially examined. When equal weights of salicylaldehyde and benzil are dissolved with aid of heat and the solution saturated with gaseous ammonia, a lemon-yellow, crystalline compound is separated, which has the composition $C_{28}H_{24}N_2O_4$, and treatment of this with dilute HCl in sealed tubes at 210° C. yields a new base, dihydroxystilbenediamine. The original compound is dibenzoyldihydroxystilbenediamine, a substitution product of this base, and was subsequently obtained by synthesis.
(*J. Ch. Soc.*, **265**, *672.*) A. A. B.

On the Introduction of the Azo-Group, in the so-called Aromatic Para-compounds. P. GRIESS.

(*Ber. d. chem. Gesell*, **338**, *1884.*) J. H. S., Jr.

On the Existence of the Trimethylen Ring. W. H. PERKIN, JR.

(*Ber. d. chem. Gesell*, *323*, *1884.*) J. H. S., Jr.

On a new Synthesis of Anthrachinone. By W. PANATOVITS.

Anthrachinone is obtained by the dry distillation of calcium phthalate.—(*Ber. d. chem. Gesell.*, *312*, *1884.*) J. H. S., jr.

ANALYTICAL CHEMISTRY.

On the Separation of I and Cl in the Dry Way. J. KRUTWIG.

On heating a dry mixture of KI and potassium bichromate (in excess), the I is completely expelled, and chromic oxide, potassium monochromate and bichromate remain behind, viz.: $6KI+5K_2Cr_2O_7=6I+Cr_2O_3+8K_2CrO_4$.

For the analysis, the KI is weighed out in a porcelain crucible. Six times as much $K_2Cr_2O_7$ is added, well mixed, with the KI, and the crucible again weighed. The crucible is then slightly heated until fumes of iodine cease to escape. This takes about 30 minutes. The crucible is weighed again, and the amount of iodine present calculated from the difference. The I may also be estimated from the amount of Cr_2O_3 formed, as follows: The melted mass is treated with water in a beaker, and heated for a short time on the water-bath. K_2CrO_4 and $K_2Cr_2O_7$ are quickly dissolved, and Cr_2O_3, which remains, is collected on a filter, washed and weighed. NaCl, on the contrary, is not attacked by $K_2Cr_2O_7$.

It is therefore possible to separate a mixture of KI and NaCl, by heating them in a porcelain crucible with $K_2Cr_2O_7$. The I is estimated as above, and Cl by acidifying the filtrate from the Cr_2O_3 with HNO_3, and precipitating with $AgNO_3$. (*Ber. d. chem. Gesell*, **341**, *1884.*) J. H. S., Jr.

Fractional Distillation in a Current of Steam as a New Means of Investigating Naphtha. (*Preliminary notice.*) RASINSKI.

Steam is conducted from a boiler to the bottom of the distilling flask (not otherwise heated). The mixture of steam and hydrocarbon vapor is dephlegmated by cooling in tubes and by washing in Mendeljeff's dephlegmators (from one to three in number). These latter are not heated. The mixed vapors coming from the last dephlegmator are collected, after condensation, in quantities of 65–70 c. c., and for each such fraction the specific gravity and the ratio of water to oil (by volume or weight) determined. The numbers thus found serve as co-ordinates for the curves representing the results of each distillation. The fractioning depends upon the peculiarity of these hydro-carbons that, when thus distilled, the ratio between the quantity of oil and of water used in distilling varies with the specific gravity of the oil. Fractions with increasing specific gravities may thus be secured without risk of decomposition. (*Jour. f. prakt. Chem.*, **29**, *41.*) F. P. V.

Determination of Morphine in Opium. V. PERGER.

On comparing the methods of E. Merk, Hager-Godeffroy, the *Pharmacopœa Austriaca*, ed. VI., and the one proposed by himself, the author finds most surprising differences in results obtained. The author's method is as follows : The opium is heated with barium hydrate and water, filtered, and washed with hot water. The filtrate, containing all morphine, is saturated with carbon dioxide and the whole rapidly evaporated to dryness on a water-bath. The dried mass is extracted with absolute alcohol and the alcoholic filtrate freed from alcohol by distillation. The residue is allowed to stand some time with ammonia water, brought upon a filter, washed with water containing ammonia, then with chloroform, then dried and weighed. This crude morphine may be purified by dissolving in acetic acid, adding a few drops of potassium ferrocyanide, filtering, neutralizing the filtrate with ammonia and allowing the morphine to crystallize out, collecting the crystals upon a filter, drying and weighing. (*Jour. f. prakt. Chem.*, **29**, *97.*) F. P. V.

Abstract of American Patents Relating to Chemistry.

(From the Official Gazette of the U. S. Patent Office.)

January 6th, 1885.

310,164.—Furnace for the manufacture of iron direct from the ore.—C. Adams.

310,187.—Apparatus for refining sulphur.—F. Dickert.

310,192.—Fibrous pulp roofing pile.—J. F. Edson.

The fibrous pulp is pressed in moulds and dried, after which it is impregnated with a waterproof solution, and coated with a fireproof enamel.

310,205.—Fabric for covering heated surfaces.—H. W. Johns.

Consists of ropes or rolls of fibrous materials, woven with sheets of paper, sheathing, &c.

310,275.—Colored waterproof fabric.—T. Hawley.

310,302.—Process for the separation of metals.—B. Moebius.

This is a process for refining silver by electrolysis. The alloy as an anode is subjected to an electric current in a solution of nitrate of silver, nitrate of copper, and nitric acid.

310,334.—Asbestos paper.—S. Tingley.

A sheet of asbestos paper is covered on one or both sides with thin paper, coated with a salt, which will form a glaze when heated to high temperatures.

310,339.—Method of manufacturing hydraulic grape sugar.—L. Virneisel.

Consists in forming a magma of crystals of grape sugar hydrate from starch glucose, mixing the magma with a thin solution of grape sugar and draining.

310,376.—Apparatus for the manufacture of prussiate of potash.—E. R. Carhuff.

Not intelligible without illustration.

310,403.—Evaporating pan and furnace.—C. W. Frick.

Adapted for cooking two lots of juice at one time. Not intelligible without illustration.

310,404.—Fire extinguishing compound.—F. Frohlich.

Consists of an alkaline silicate, alum, common salt, and phosphate of ammonia.

310,410.—Incaustation preventive.—H. Gyrard.

Malt sprouts are distilled and the resulting liquor is added to the water in the boiler.

310,448.—Process of and apparatus for baryta or strontia treatment of saccharine liquors, and production and recovery of these reagents.—H. Leplay.

310,454.—Metallurgical furnace.—W. McKenna.

This furnace is adapted for burning gas.

310,461.—Non-conducting composition for refrigerators.—J. M. Ordway.

Consists of a non-conducting coating of granulated cork, bark, or pith, agglommerated with water glass, in combination with a lining of fossil meal or magnesia.

310,486.—Producing gelatinous and printing plates.—W. Woodbury.

Consists in producing a photographic design in gelatine plates, then drying and mounting them on solid backs, and coating the surface with tin foil. By subjecting this to pressure a metallic printing surface is obtained, which may be electroplated.

310,487.—Apparatus for the manufacture of gas.—A. L. Allen.

A current of steam or super-heated steam is passed continuously through a mass of highly heated carbon of gradually increasing temperature, and finally through one or more independent masses of incandescent carbon of equal temperature with the final temperature of the decomposing retort.

310,497.—Process of and apparatus for the fractional distillation of petroleum.—R. Dean.

Not intelligible without drawing.

310,500.—Process of constructing lead vessels.—H. Egells.

The body of the vessel is constructed of one of the harder alloys of lead—such as type metal—and lead is directly fused upon the surfaces to be exposed to corrosive action.

310,533.—Apparatus for the electrolytical separation and deposision of metals.—B. Moebius.

310,544.—Apparatus for manufacturing flexible roofing material.

January 13th, 1885.

310,550.—Lime paint.—W. J. Adams and W. R. Polk.

A lime paint consisting of lime, salt, alum, syrup, and a gluey substance.

310,560.—Preparation of a fluid for making water repellent fabrics.—A. B. Conger.

Caoutchouc or other elastic gum is treated with gas of nitric or muriatic acid, and dissolved with paraffin or wax in naphtha or other mineral oil.

310,594.—Process of manufacturing asphaltic powder suitable for paving.—H. Kettmann.

Asphaltic concretes are made by mixing asphalum or bitumen, with pulverized material, while the latter are suspended in water.

310,604.—Manufacture of isatins and substituted isatins.—P. J. Meyer.

The isatins and substituted isatins are obtained from dihalogenized acetic acids, their salts, amides, ethers, and aldebydes or from amines, by directly fusing or boiling their solutions, and treating the product with a strong acid.

310,619.—Ore furnace.—E. F. Russell.

310,625.—Composition for granite or gravel roofing.—F. Vaughan.

A mixture of linseed oil, resin, pitch, beeswax, and carbolic acid.

310,744.—Gas retort.—A. C. Swain.

Not intelligible without drawing.

310,753.—Treatment of yucca or "sotal" fibre for the production of paper pulp or stock and obtaining by products therefrom.—G. B. Walker.

The fibre is steeped in acid sulphite liquors, then digested in borax liquor and sulphurous acid, and finally washed in a weak soda solution.

310,759.—Limekiln.—A. B. Weeks.

310,804.—Glass furnace.—J. V. Ebel.

January 20th, 1885.

310,887.—Fire-Extinguishing Compound.—J. M. Giblin.

A vessel of glass, or analogous frangible material, containing liquefied sulphurous oxide.

310,888.—Fire-Extinguisher.—J. M. Giblin.

310,889.—Fire-Extinguishing Compound.—J. M. Giblin.

Consists of a solution of sulphurous oxide, in an alkaline liquid.

310,899.—Plastic compound suitable for molding into various useful articles.—M. Mackay.

Consists of lac, gum sandarac, rosin, ivory, black, and asbestos.

310,901.—Process of making ferronickel and ferrocobalt.—H. Marbeau.

Mats or pigs of these metals are fused with potassium ferrocyanide or cyanide and one of the oxides of manganese, and at the moment of casting a small quantity of aluminium is added.

310,925.—Thermometer.—H. Weinhagen.

A dark, light-absorbing shield is placed behind the mercury column.

310,957.—Manufacture of compounds to be used for insulating.—A. Muirhead.

Consists of a mixture of purified paraffin wax or other hydrocarbons and gutta percha.

310,963.—Spirit Condenser.—F. Sonier.

Not intelligible without the drawings.

311,013.—Ice machine.—J. C. Kitton.

311,028.—Refrigerating machine.—A. Osenbrueck.

311,058.—Ore roasting furnace.—J. F. Carter.

311,062.—Refrigerating apparatus.—N. W. Condit, Jr., and T. Rose.

311,118.—Manufacture of amber varnish.—J. W. Goussen.

The amber is ignited upon a wire cloth, and allowed to drip through the cloth, after which it is boiled with the oil, and finished in the usual manner.

311,124.—Process and apparatus for making gas.—J. Hanlon.

312,125.—Process and apparatus for making gas.—J. Hanlon.

Illuminating gas is made by continuously heating the generating retorts by the combustion of gaseous products from a producer, and such products, after partial combustion, are used for alternately heating internally two fixing-chambers. Gas is generated continuously in the retorts, by passing superheated steam down through a body of charcoal, where it is decomposed, passing the resulting gas up through the vaporizing chamber, into which oil is admitted, and then passing the mixed gas and oil vapor through one of the heated fixing chambers, where they are converted into a fixed gas. One of the fixing chambers is being heated up while the other is used for fixing gas.

311,135.—Recovering rubber from rubber waste.—C. I. McDermott.

The fibre is removed from fibrous rubber waste or scrap, by treating with dilute acid, or acid containing materials for generating nascent chlorine.

311,156.—Manufacture of artificial stone compositions.—C. I. Walker.

Lime and silica are ground to powder and intimately mixed. The lime is then slaked and sand or gravel is added.

January 27th, 1885.

311,203.—Composition of matter for water proofing.—I. V. Reagles.

The composition consists of wood alcohol, castor oil, gum camphor, pyroxiline, and gum shellac.

311,223.—Filtering paper.—G. H. Billings, E. W. Carter, and D. R. Smith.

The sheets of filtering paper have a fibrous material embedded in the center.

311,257.—Filtering apparatus.—C. W. E. Piefke.

311,258.—Amalgamator.—D. S. Randolph.

311,271.—Disinfecting apparatus.—R. Thayer.

311,287.—Plastic cement mixture for non-conducting coverings for boilers, &c.—D. A. Brown.

The covering consists of infusorial earthy lime and asbestos.

311,294.—Process of surface coating iron.—P. H. Conradson.

The iron is exposed to sulphur vapor in a muffle in an atmosphere of carbon dioxide, at a temperature of 500°-600° C. Sulphuretted hydrogen may be substituted for the sulphur vapor.

311,360.—Process of disinfecting baled rags.—S. W. Parker and H. Blackman.

A perforated nozzle is inserted into the bale, and the disinfectant is cause to pass through the nozzle and out through the perforations thereof.

311,401.—Paint.—W. H. Wilber.

A priming paint composed of liquid asphaltum, rosin, linseed oil, turpentine or naphtha and white lead.

311,403.—Furnace for generating illuminating gas.—S. J. Anthony.

Steam is superheated and decomposed in the lower part of the generator, and the resulting gas is carburetted and fixed in the upper part thereof. Perforated tile or fire brick strainers in the upper part of the furnace serve to heat the gas and to remove from it the lamp black and tarry matter.

311,417.—Method of manufacturing maltose.—L. Cuisinier.

The amylaceous material is reduced to a pulp, and then water containing an infusion of malt is added. The mass is gradually heated to about 80° C. for about 1 hour, and then digested under a pressure of 1-2 atmospheres for about half an hour.

311,441.—Gas generator.—L. Mond.

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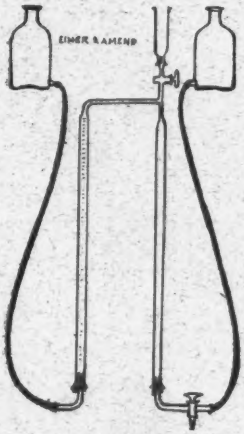

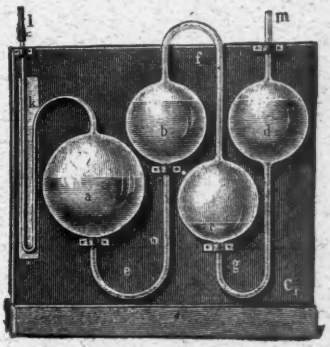

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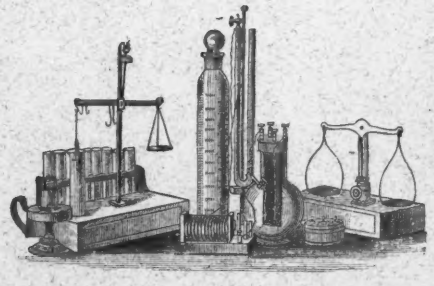

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